

## **Workshop Report**

### **Hands-on Computer-assisted X-ray Microtomography (micro-CT) Workshop (Naval Research Laboratory, Stennis Space Center: April 21 – 23, 2004)**

*Naval Research Laboratory Code 7430*

*April 30, 2004*

#### **Summary**

Hands-on micro-CT Workshop was held April 21–23, 2004 at the Naval Research Laboratory, Stennis Space Center, Mississippi, USA. The objective of workshop was to initiate and promote collaborative research associated with NRL's new, high-resolution (to  $\sim 10\ \mu\text{m}$ ) industrial Computer-assisted Tomography (CT) system, HD-500. Specifically, the workshop was designed to familiarize the potential collaborators with the system's capabilities and limitations. The micro-CT system was a recent (FY03) major capital purchase at NRL.

Twenty-three scientists and engineers representing eleven institutions and companies participated in the workshop. Abraham Grader (Penn State), the foremost expert in the scientific applications of micro-CT, led the discussions to explore a variety of scientific problems that can be solved by micro-CT. Allen Reed, NRL's micro-CT expert, led the hands-on experiments with samples provided by the participants. Representatives from imaging software companies were present to help participants explore the various data manipulation options.

The success of each hands-on experiment during the workshop was ensured by NRL's months-long preparation prior to the workshop. The preparation, led by Dr. Reed, included communications with workshop participants, sample acquisition, and preliminary test experiments. In addition, certain experiments required custom-built sample holders and *in-vitro* experimental devices, which were designed and built specifically for the micro-CT sample stage with the aid of frequent communications between participants and Dr. Reed. At the end of the workshop, we were able to furnish all participants with the high resolution CT data of their specimens, as summarized in the next section. Some of the data sets obtained during the workshop are already publication quality whereas other data sets will be used to refine the plans and designs for further experimentations and CT scanning.

The workshop was beneficial to both outside and NRL participants. The participants had a chance to closely interact with Drs. Grader and Reed using their familiar specimens as the media. They gained in-depth understanding of the micro-CT system in the context of their own samples, which should foster further collaborative research ideas. In addition, the workshop brought up and defined new scientific challenges and demands for NRL's micro-CT facility to pursue in order to make the facility truly recognized as the leading CT facility for science and technology applications.

The success of workshop has put NRL's micro-CT facility in a good position to reach out to the greater scientific and technology community. The workshop results will be summarized and submitted for publication on *EOS*. Post-workshop visits to funding agencies are planned by

Allen Reed and Kevin Briggs. Based on preliminary results from the workshop, multi-institution joint proposals are planned.

## Summary of Experiments

### *Experiment 1 “Shear band in sands”*

**21 APR 0900 – 1200**

**Work Group 1: Amy Rechenmacher (JHU), Khalid Alshibli (LSU), and Andrei Abelev (NRL/USM)**

Accurate mapping and qualitative description of shear bands, or zones of localized deformation, is important in describing the behavior of materials during the initiation of failure and beyond. CT scans of a resin impregnated sand specimen were performed at the following energy settings:

- Acceleration potential: 150 kV
- Current: 40  $\mu$ A

producing the resolution in the X-Y-Z coordinates of 15, 15, and 30  $\mu$ m, respectively. The specimen scanned was made of uniform sand with the mean particle size of 220  $\mu$ m.

The existence of shear band was not at all obvious under visual examinations. However, our micro-CT made possible the differentiation between the two-phase material. Two distinct planes were identified with the micro-CT and the locations of the bands were made apparent with the Amira software from TGS. The identification was performed by mapping the interconnected areas of unusually large voids between the granular matrix, characteristic of a developed shear band. The following two images (Figure 1) show the results. Two planes, marked by blue and red interconnected pores, are apparent.

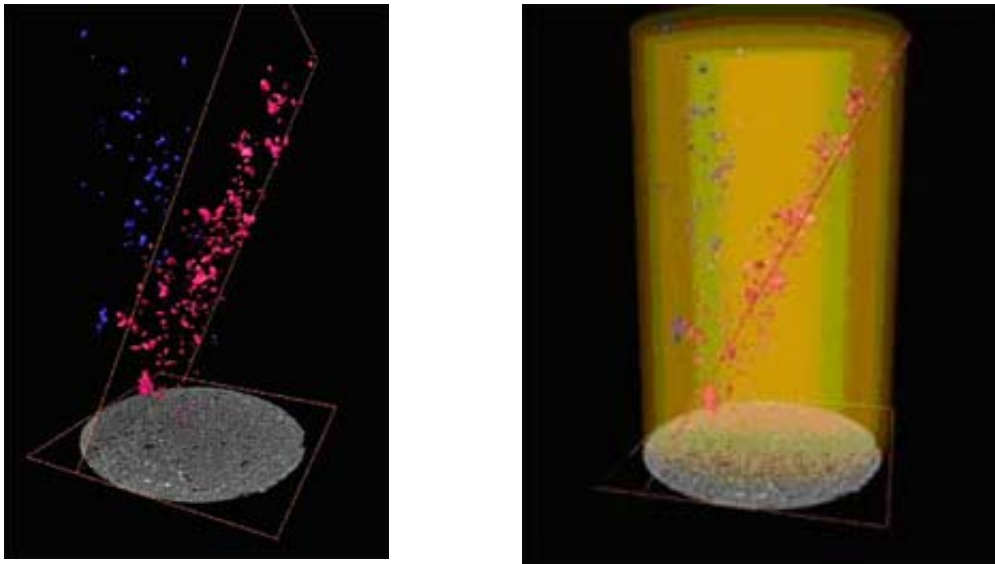


Figure 1. NRL’s micro-CT was able to identify shear bands in the granular sand sample.

The movie clips (Appendices I and II) clearly show the planar structure of the two shear bands identified.

## ***Experiment 2 “Multiphase flow”***

**21 APR 1230 – 1700**

**Work Group 2: Markus Hilpert (JHU), Karsten Thompson (LSU), Clint Wilson (LSU), and Allen Reed (NRL)**

Quantifying the constituents multiphase flow systems is necessary to gain understanding about the processes that occur in the beach swash-zone, in contaminated sediments, and in petroleum reservoir rocks and sands. The ability to differentiate fluid and solid phases within a sediment, or rock, system (Figure 2) is key to quantifying relative permeability. It will also help facilitate understanding about how drainage and imbibition influence sediment properties and transport, mediate contaminants in sediments, and to increase petroleum extraction efficiencies.

The primary purpose of the experiment was to generate images for lattice boltzmann and network modeling of the fluid mechanics that operate in these systems. Future work will be to evaluate contaminant mitigation, imbibition and drainage in glass beads and marine sand (Al-Raoush et al., in press; Hilpert and Miller, 2001; Thompson and Fogler, 1996;), from pore scale network data.

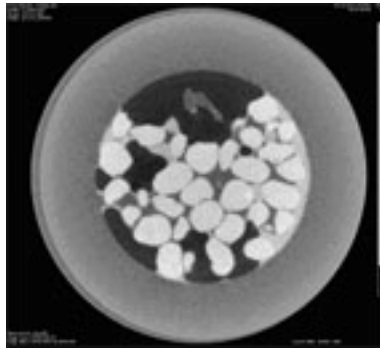


Figure 2. A water-saturated quartz sand (Light gray oblate shapes) system was infiltrated with oil (light gray pendular rings at the edges of dark blobs) and air (dark blobs). Water is medium gray and slightly darker than the surrounding plastic (i.e., acrylic) tube.

## ***Experiment 3 “Gas hydrate structure and constituents”***

**22 APR 0900 – 1300**

**Work Group 3: Bill Winters (USGS), Dawn Lavoie (USGS), Fritz Abegg (GEOMAR), and Warren Wood (NRL)**

Methane hydrate is an ice-like combination of methane and water that forms in natural sediment, but is stable only under high pressure and/or low temperature, and is therefore found on nearly all continental margins in more than about 400 meters of water or close to the land surface in permafrost settings. When brought to standard temperature and pressure, small samples of methane hydrate dissociate to methane gas within seconds, making it very difficult to study in a conventional CT laboratory setting. To try to overcome this challenge, NRL built a simple cryogenic stage (Figure 3) that consists of a container of liquid nitrogen, and a small tube or chimney within which the sample is placed. The narrow chimney focuses cold nitrogen gas around the sample, thus keeping the hydrate in a stable state, while minimizing the amount of material around the sample that could attenuate the x-ray stream and degrade the scan. Samples can be kept stable for the many tens of minutes required for a detailed scan.



Figure 3. Cryogenic stage for in-vitro methane hydrate tomography attached to the CT sample stage.

The cryogenic stage was successfully used to scan a sample of methane hydrate that has been stored at liquid nitrogen temperatures since October of 2003. The sample was acquired by Dr. Fritz Abegg in the southern Gulf of Mexico in the proximity of bacterial mats on the seafloor. The white circle in the three images of Figure 4 is the aluminum sample tube (inside diameter of 14 mm), the white spiral and specks in the image are calcium carbonate shell, and the dark areas are empty space (air in this in vitro sample). The lighter gray is the fine grained sediment matrix and the darker gray is either water ice or methane hydrate or both. The two materials have similar densities and very likely similar x-ray attenuation characteristics. Although no methane hydrate can be identified unequivocally, this preliminary workshop experiment showed that hydrate laden sediments can be accurately scanned at very high resolution (several times that of a medical CT scan), and that the high resolution can be maintained even through a 1 mm thick aluminum sample tube.

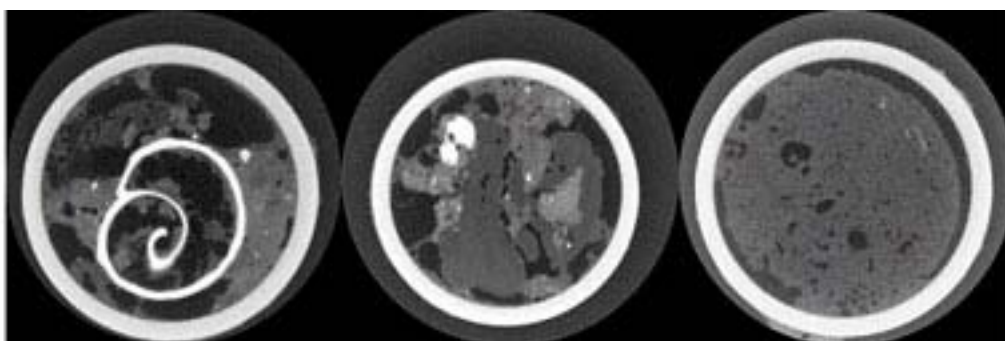


Figure 4. Representative horizontal renditions of the cryogenic methane hydrate scan.

To distinguish the methane hydrate from water is far easier if the water is kept in the liquid phase, but this would require maintaining the sample at pressures of many tens of atmospheres. Plans are currently underway to perform similar scanning experiments using the Department of Transportation approved pressure vessels that are the current standard for transporting methane hydrate. Within the pressure vessels methane hydrate can be kept at in situ pressures and temperatures, but it is not yet known how the denser and thicker walls of the pressure vessels may degrade the CT scan. It was suggested that a pressure vessel made of fiberglass to take up the radial stress, combined with a relatively thin aluminum tube to take up the longitudinal stress, would be strong enough to maintain methane hydrate at in situ pressures while minimizing x-ray attenuation.

A technique for scanning samples under pressure would also be of significant value in collaborations with the USGS's GHASTLI (gas hydrate and sediment testing laboratory instrument), which is a unique triaxial cell in which pore pressure, confining pressure, and vertical stress can all be controlled independently. The engineers at USGS led by Dr. Bill Winters have more than a decade of experience regarding the acoustic, elastic, thermal, electrical properties of sediment, fluid, and gas samples under controlled pressure and temperature. A high resolution image of where and how bubbles and hydrate form under various in situ conditions is the most obvious component GHASTLI lacks, so a collaboration involving the two state-of-the-art systems promises to be especially fruitful.

#### ***Experiment 4 “Volume inhomogeneities”***

**22 APR 1330 – 1700**

##### **Work Group 4: Dajun Tang (UW) and Kevin Briggs (NRL)**

Characterization of fluctuations in sediment bulk density is essential to modeling the acoustic scattering from sediments. The volume inhomogeneities created by sedimentological, biological, and physical processes are present in the sediment volume and have physical dimensions that make interaction with high-frequency sound likely. Thus, methodology to map the size, shape, and orientation of inhomogeneities within the sediment volume is of significant interest to acoustic modelers. Fortunately, CT provides a non-destructive method of measuring the dimensions and density of potential acoustic scatterers.

Besides mapping the calibrated density fluctuations within a sediment core sample in 3D, CT can be used to isolate and characterize specific inhomogeneities embedded in the sediment matrix. For instance, shells of previously live marine organisms as well as fragments of shells can be imaged for the purpose of ascertaining their size, volume, and orientation. The goals of this experiment were to image a gastropod shell within a matrix of simulated sediment (glass beads) and to image the larger, complex structure of a sand dollar test and calculate its solid volume. As evidenced by the images in Figures 5 and 6, these goals were impressively achieved. The solid volume of the sand dollar was approximately 6 cm<sup>3</sup>. Appendix III shows the movie rendition of the internal structure of sand dollar.

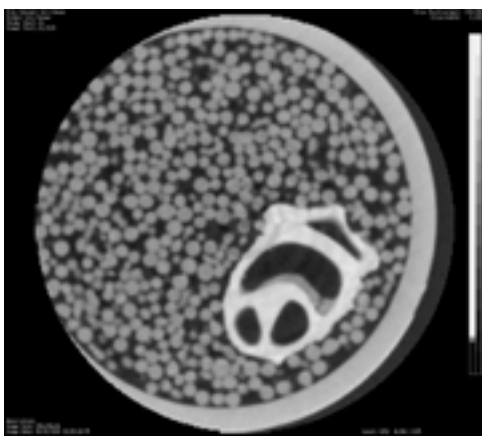


Figure 5. Gastropod shell embedded in glass beads



Figure 6. Sand dollar.

## ***Experiment 5 “Bubble growth in mud”***

**23 APR 0900 – 1630**

**Work Group 5: Bernie Boudreau (Dalhousie), Chris Algar (Dalhousie), Allen Reed (NRL), and Yoko Furukawa (NRL)**

Diagenetic methane bubbles are commonly found in fine-grained littoral sediments and pose problems for the interpretation of seafloor acoustic signals. Dr. Boudreau and his colleagues have been investigating the growth mechanisms of diagenetic methane bubbles using physics-based analytical computational models (Boudreau et al. 2001; Gardiner et al. 2003). They are also monitoring the physical properties of methane-rich mud off the coast of Nova Scotia.

The primary purpose of the hands-on experiment was to ground-truth the computational modeling results: diagenetic methane bubbles in cohesive sediments grow by fracturing, thus the bubbles are shaped as oblique spheroids rather than spheres. In order to accomplish the goal, Dr. Boudreau and his Dalhousie colleague Dr. Bruce Johnson had modified their custom-built bubble growth experimental device fitted with a micropressure gage so that the experiments can be conducted on the CT sample stage while pressure changes are being monitored and CT data are being collected (Figure 7).



Figure 7. Bubble growth experimental device adapted for the in vitro experiments on CT sample stage. (Device and image courtesy of Drs. Bruce Johnson and Bernard Boudreau, Dalhousie University)

The goal was accomplished as shown in Figure 8. The bubble grew by fracturing the matrix mud, occupying a thin disk with the diameter of  $\sim 20$  mm and thickness of  $\sim 0.7$  mm. The geometrical properties obtained here will be compared to the model geometry obtained by Dr. Boudreau's model. Appendix IV shows the movie rendition of the same bubble.

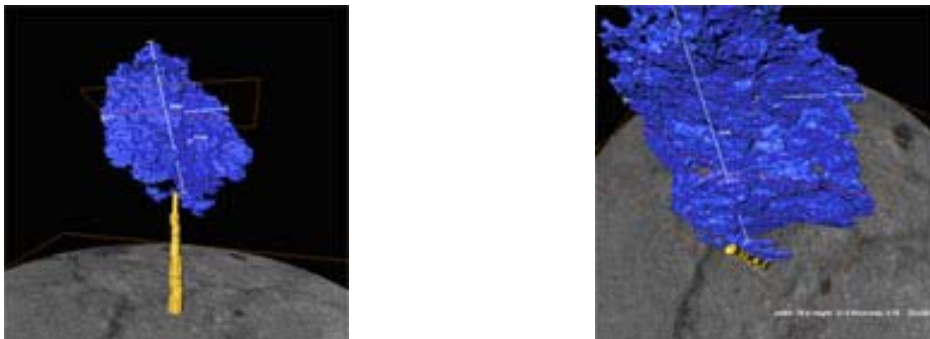


Figure 8. The CT image of bubble grown in a cohesive sediment core sample from Nova Scotia. The capillary tube used to deliver gas is rendered in gold whereas the bubble, grown by a thin fracture, is represented in blue. The bubble has the diameter of  $\sim 20$  mm and thickness of  $\sim 0.7$  mm.

The future wish list for this Work Group includes the coordinated characterization of bubble geometry and growth pathways together with the physical properties pertinent for the growth model parameterization.

### ***Parallel Workshop “Data rendering options”***

**21 – 23 APR**

**Data rendering software representatives: Bob Kehl (Kehl Co.), Hauke Bartsch (TGS), and Richard Johnson (TGS)**

Throughout the workshop, representatives from the imaging/data rendering software companies were on hand to help participants retrieve necessary information from the raw CT data generated during the hands-on experiments. Most participants’ primary interests were to obtain geometrical properties (e.g., volume, dimension) at this time. However, the participants have also learned that other physical properties, namely those related to density, can be rendered from raw CT data.

### **Acknowledgment**

The workshop was made possible by NRL Code 7000 B&P Fund (Dr. Eric Hartwig, Associate Director of Research). Participations of international scientists were supported by Office of Naval Research International Field Office, under its VSP program (CDR Christopher Butler, Associate Director).

## Participants

NAME	AFFILIATION	E-MAIL
Friedrich Abegg	GEOMAR	fabegg@geomar.de
Andrei Abelev	NRL	abelev@nrlssc.navy.mil
Chris Algar	Dalhousie University	calgar@dal.ca
Khalid Alshibli	LSU	alshibli@lsu.edu
Hauke Bartsch	TGS Inc.	hauke@tgs.com
Bernard Boudreau	Dalhousie University	bernie.boudreau@dal.ca
Kevin Briggs	NRL	kevin.briggs@nrlssc.navy.mil
Yoko Furukawa	NRL	yfurukawa@nrlssc.navy.mil
Avrami Grader	Penn State	grader@pnge.psu.edu
Markus Hilpert	Johns Hopkins University	markus_hilpert@jho.edu
Richard Johnson	TGS Inc.	richardj@tgs.com
Gene Kelly	NAVO	kellyg@navo.navy.mil
Bob Kehl	Kehl Company	kehlco@kelco.com
Dawn Lavoie	USGS	dlavoie@usgs.gov
Allison Moreland	NAVO	morelanda@navo.navy.mil
Amy Rechenmacher	Johns Hopkins University	alr@jhu.edu
Allen Reed	NRL	allen.reed@nrlssc.navy.mil
Michael Richardson	NRL	mrichardson@nrlssc.navy.mil
Dajun Tang	University of Washington	djtang@apl.washington.edu
Karsten Thompson	LSU	karsten@lsu.edu
Clint Wilson	LSU	cwilson@lsu.edu
Bill Winters	USGS	bwinters@usgs.gov
Warren Wood	NRL	wwood@nrlssc.navy.mil

## References

- Al-Raoush, R., K. E. Thompson, and C. S. Willson, Comparison of network generation techniques for unconsolidated porous media systems, *Soil Sci. Am. J.*, in press.
- Boudreau, B. P., B. S. Gardiner, and B. D. Johnson. 2001. Rate of growth of isolated bubbles in sediments with a diagenetic source of methane. *Limnology and Oceanography* **46**: 616-622.
- Gardiner, B. S., B. P. Boudreau, and B. D. Johnson. 2003. Growth of disk-shaped bubbles in sediments. *Geochimica Et Cosmochimica Acta* **67**: 1485-1494.